Multiscale Characterization

Characterization of Composite Layers

- Graded Ceramic/Metal Matrix Composites
- Polymer Matrix Composites
- Local Strain Fields/Damage Initiation

Interfaces and Bonded Joints

- Thermal Impedance
- Interfacial Delamination

Structural Performance

- Impact Response
- Vibration Analysis

Nancy R. Sottos
University of Illinois

Characterization Scales

nm

reinforcement/ matrix interface

µm

microvascular channels

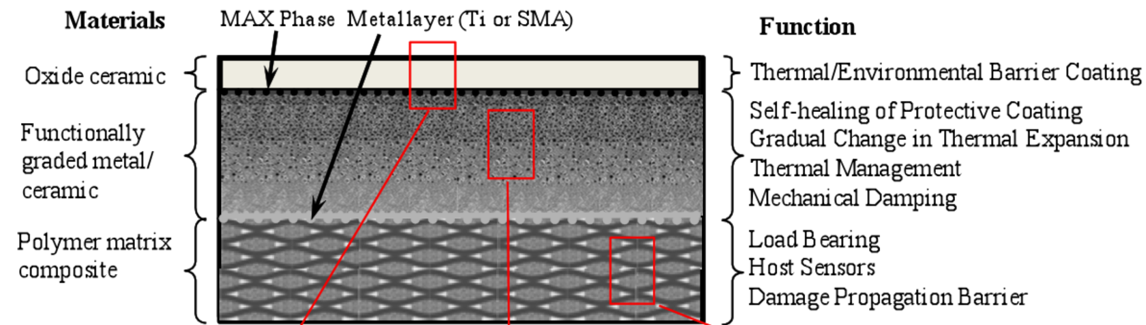
bonded interfaces

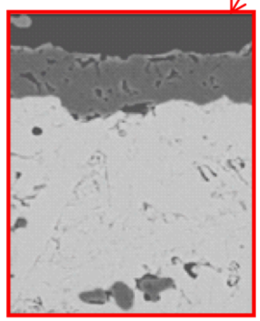
mm

composite layer properties

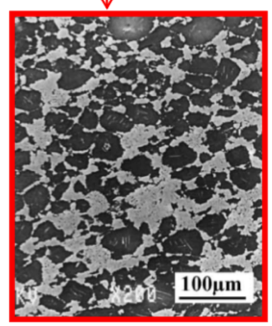
structural response

cm

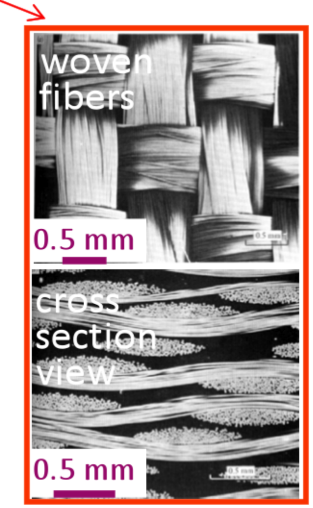




SEM micrograph of 15 µm thick protective Al_2O_3 surface layer formed after 10,000 heating cycles of Ti_2AlC [Ref]



SEM of Ti_2AlC (light) + γTiAl (dark) as an example of MAX phase composite. [Ref]

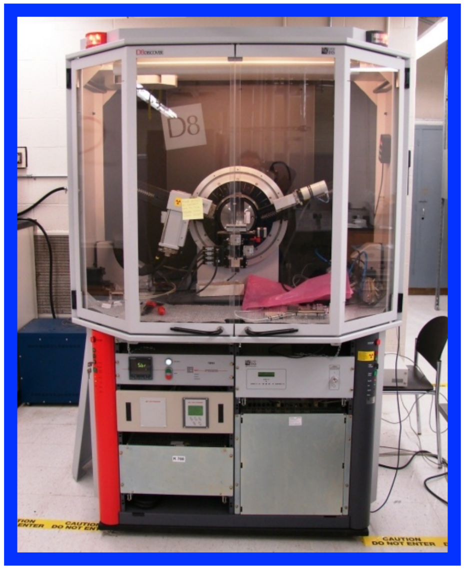


Characterization of Composite Layers

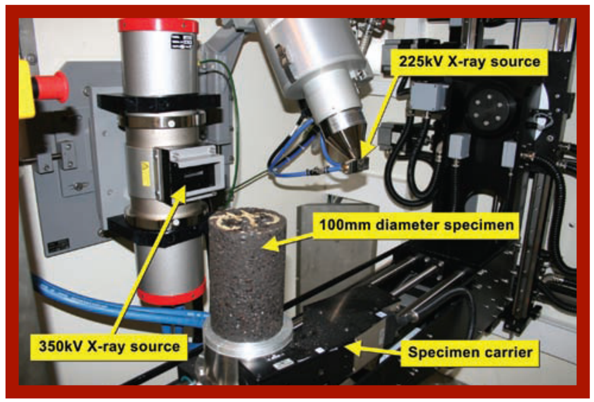
- **Graded Ceramic/Metal Matrix Composites**
  - **Ibrahim Karaman (TAMU), Miladin Radovic (TAMU), Ozden Ochoa (TAMU)**
- **Polymer Matrix Composites**
  - **Scott White (UIUC), Nancy Sottos (UIUC), Zoubeida Ounaies (TAMU)**
- **Local Strain Fields/Damage Initiation**
  - **Nancy Sottos (UIUC) , Scott White (UIUC), Ibrahim Karaman (TAMU)**

Structural Characterization of GCMeCs

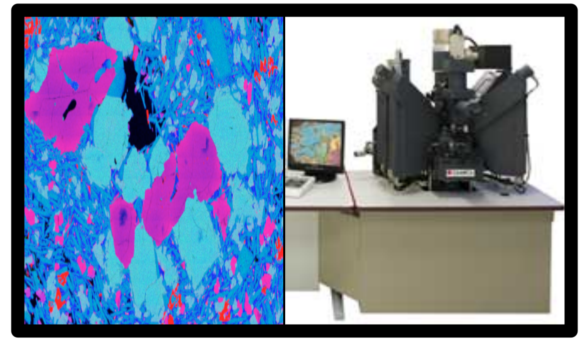
High Temperature X-Ray Diffractometer (up to 1500K)



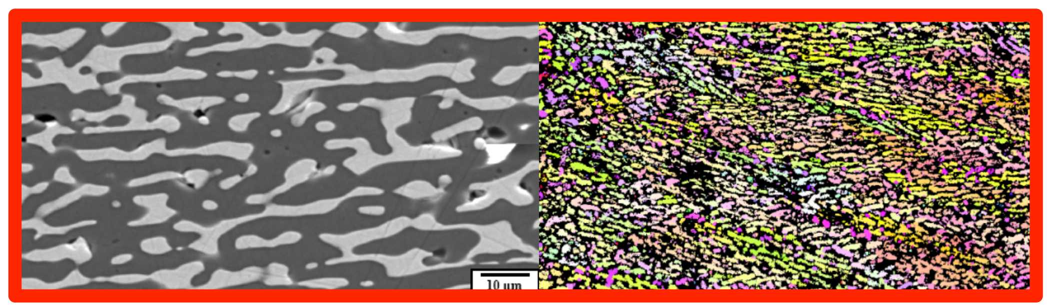
Micro CT for non-destructive characterization of metallic and ceramic phases and porosities



Electron Microprobe Analyzer and Wave Dispersive Spectroscopy (WDS) to study compositional variations across interfaces



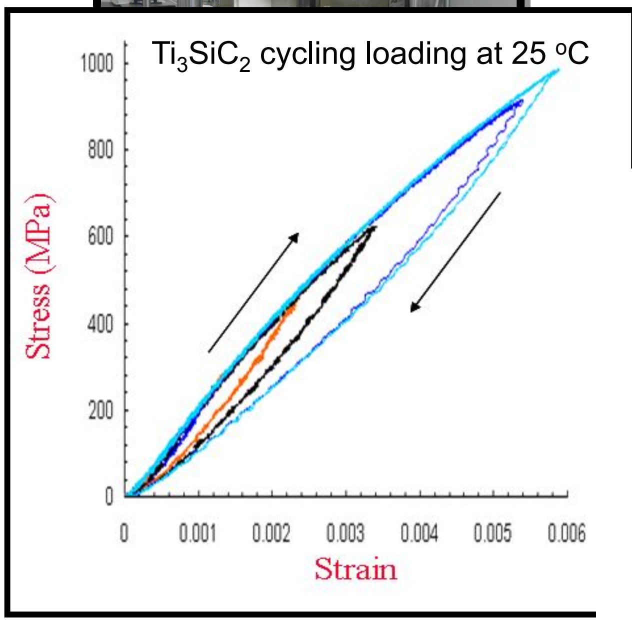
SEM and Orientation Imaging Microscopy (OIM) for phase morphology, distribution, and texture



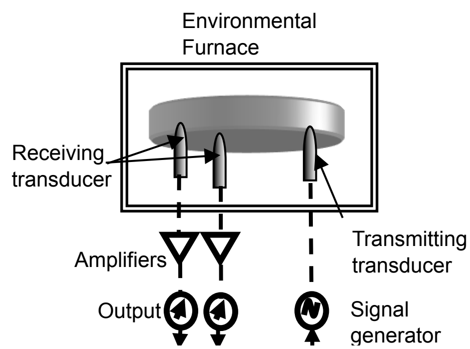
Hermetic, beryllium dome high temp heating stage under 6×10^{-7} mBar vacuum

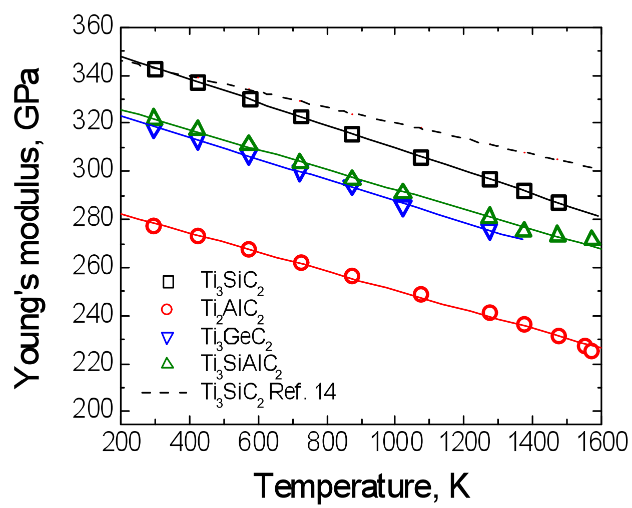
Thermomechanical Chracterization of GCMeCs

Mechancial Testing (up to 1700 °C)

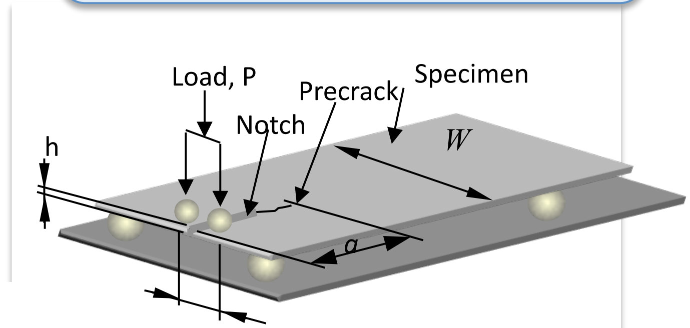


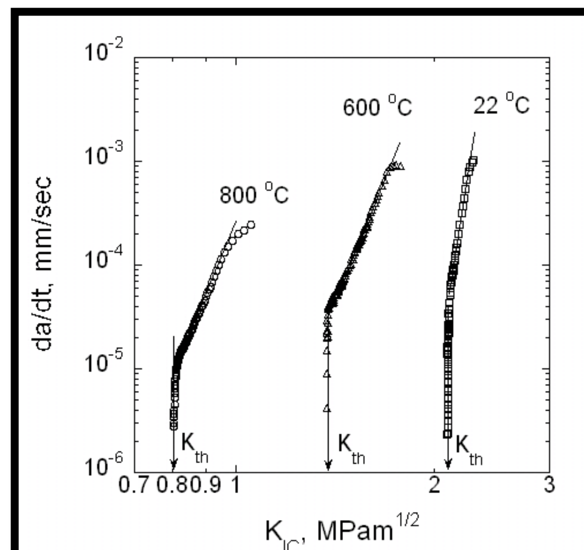
Elastic and Shear Moduli
Resonant Ultrasound Spectroscopy (up to 1300 °C)





Fracture Toughness
High Temp Double Torsion



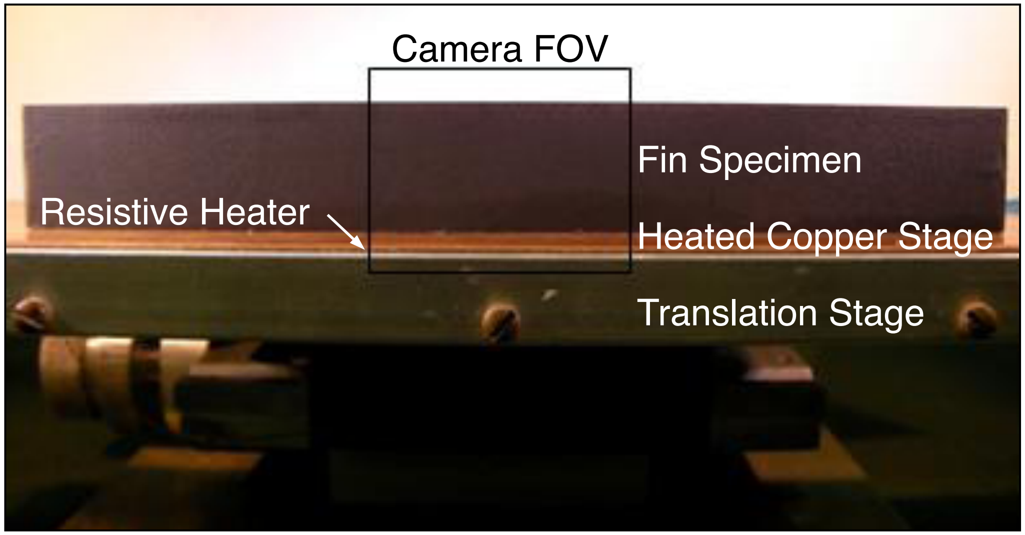


PMC Characterization

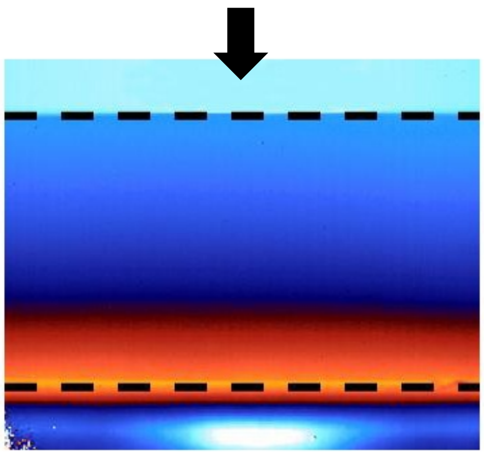
- **A full suite of experimental techniques will be used to aid manufacturing development studies and mechanical/thermal performance assessment**

Material	Experimental Technique	Characterization
Matrix Resin	Rheology	complex viscosity
	DSC	cure kinetics
	DMA	mechanical properties
Composite material	DSC	cure kinetics
	DMA	mechanical properties
	Optical/electron/fluorescent microscopy	material architecture
	Micro-CT	material architecture
Microvascular composite	IR imaging	surface temperature field
	Fluorescent microscopy	internal fluid temp
	Micro-CT	network architecture
	Micro-PIV	flow characteristics

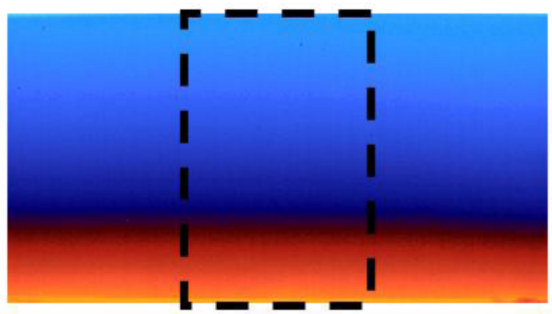
Thermal Performance Studies

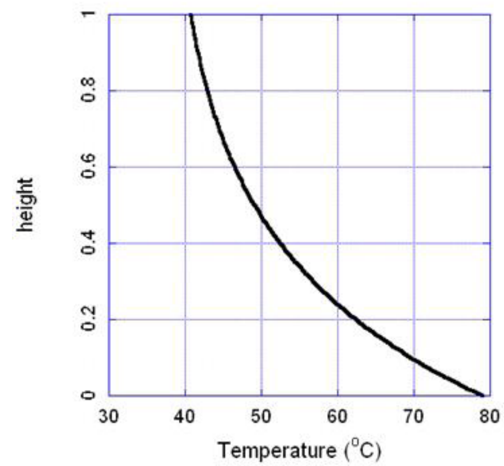


DeltaTherm 1560 infrared camera
(256 x 320 pixels Indium Antimonide
infrared detectors)



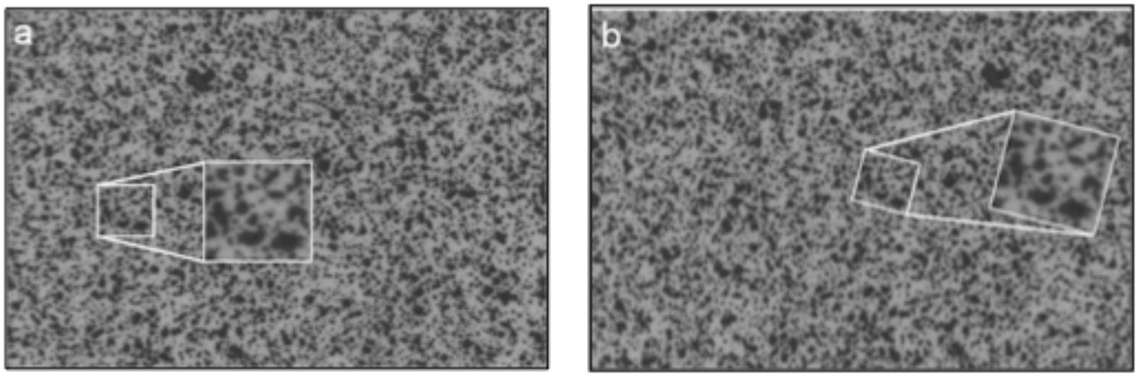
Active Cooling





Deformation/Strain Measurements

Digital Image Correlation (DIC)

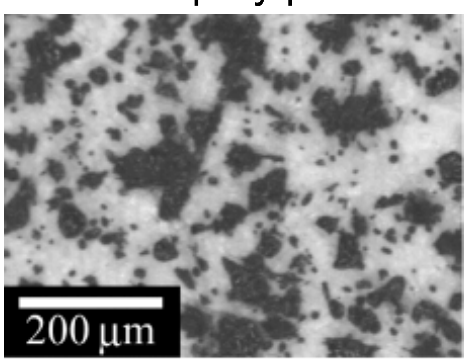


$$x'_{q'} = x_q + u_{x_p} + \frac{\partial u_{x_p}}{\partial x}\Delta x_q + \frac{\partial u_{x_p}}{\partial y}\Delta y_q$$

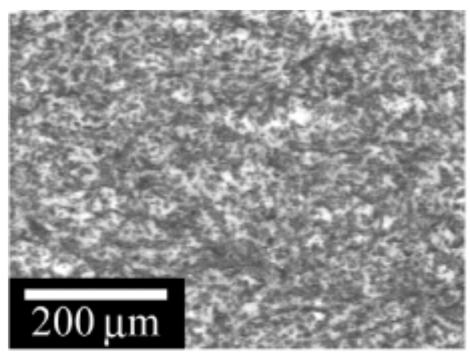
$$y'_{q'} = y_q + u_{y_p} + \frac{\partial u_{y_p}}{\partial x}\Delta x_q + \frac{\partial u_{y_p}}{\partial y}\Delta y_q$$

Can control resolution through speckle pattern:

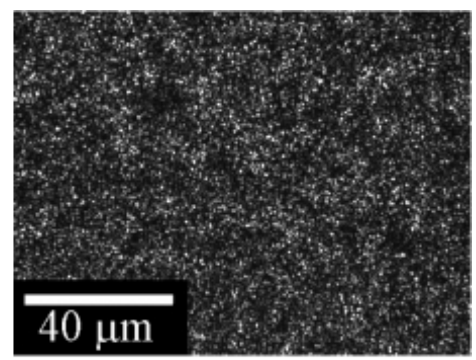
Macroscale: ±25 μm
black spray paint



Microscale: ±1 μm
air-brushed pattern

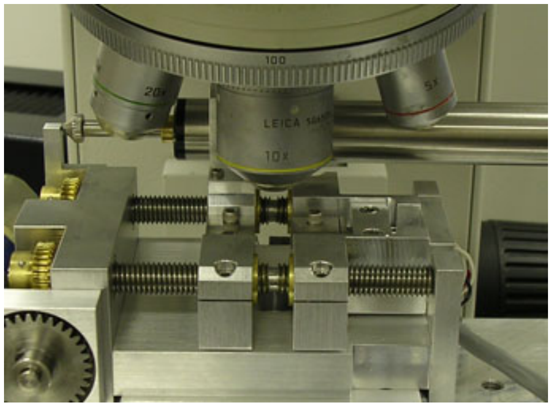


Nanoscale: ±10 nm
fluorescent nanoparticles

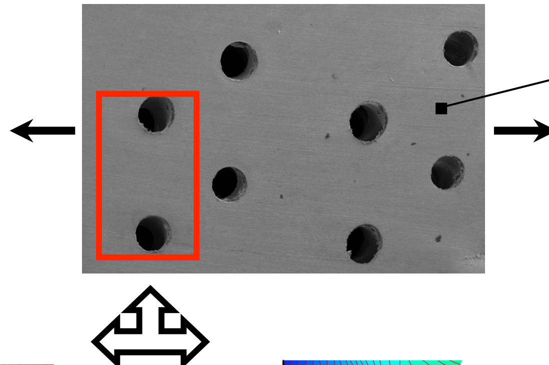


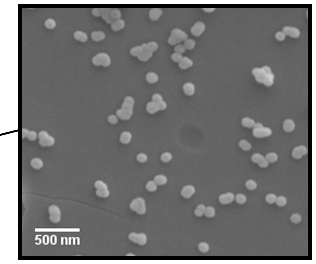
Localized Strain Measurements

Strain concentrations due to vascular networks for active cooling

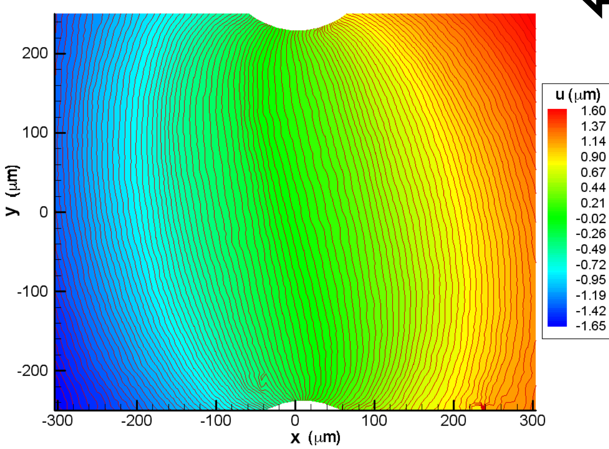


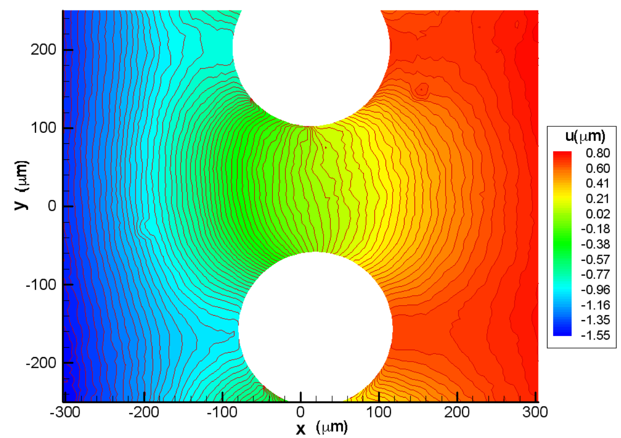
μVAC cross-section exposing channels





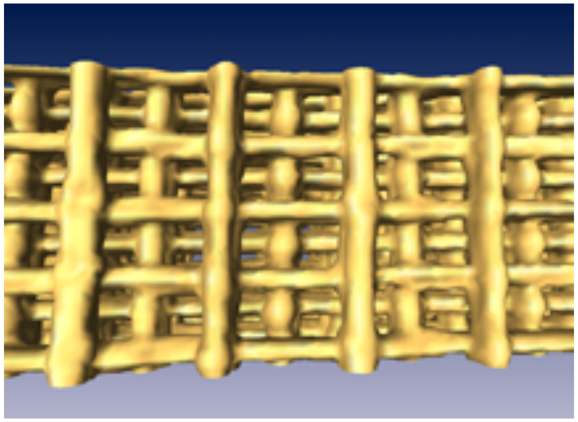
SEM of nanoparticles on sample surface



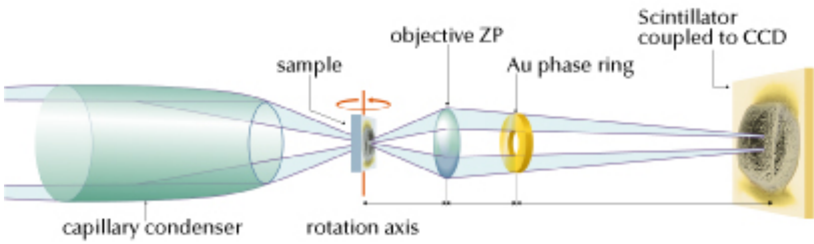


Micro and Nano X-Ray Tomography

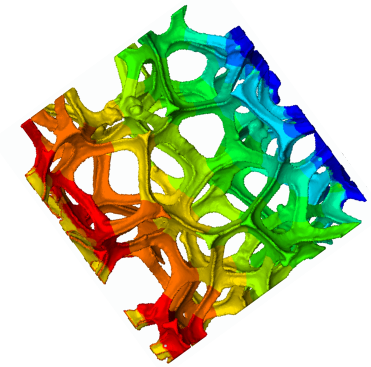
MicroCT reveals material architecture



Lens based x-ray microscopy



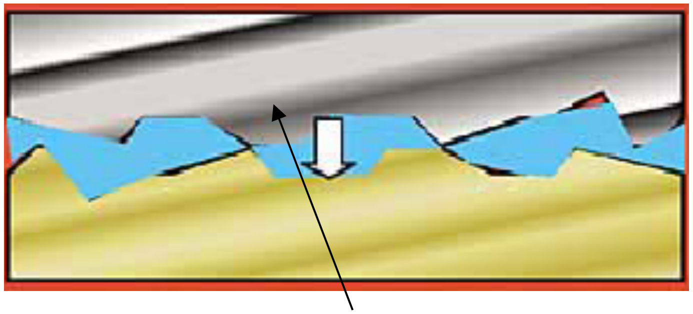
FEA models will be based on the microscopy and micro-CT observations of functionally gradient surfaces.



Interfaces and Bonded Joints

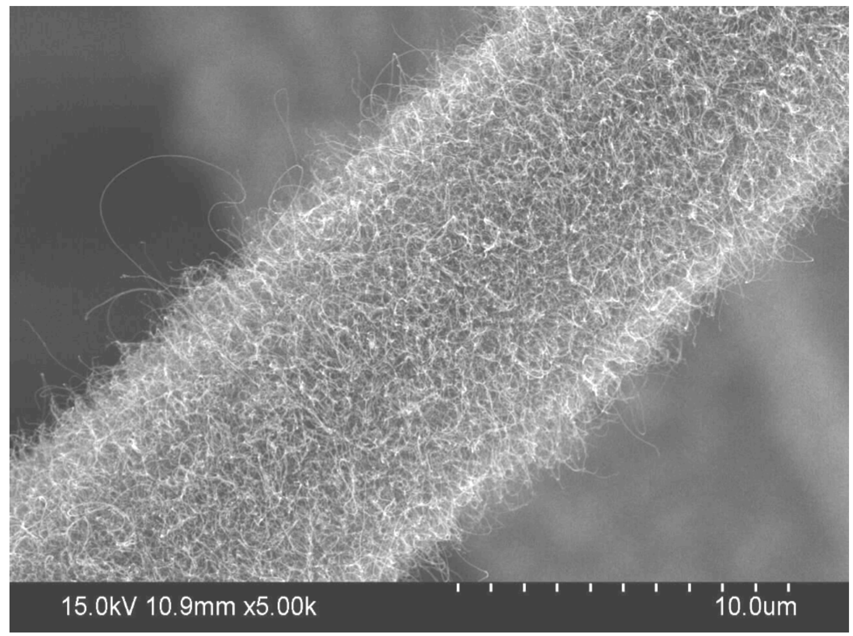
- **Thermal Impedance**
  - Khalid Lafdi (UDRI)
- **Interfacial Fracture**
  - Zoubeida Ounaies (TAMU), Ozden Ochoa (TAMU)
- **Dynamic Shear Strength**
  - Nakhiah Goulbourne (UM/VT)

Thermal Interface Materials



Thermal interface material

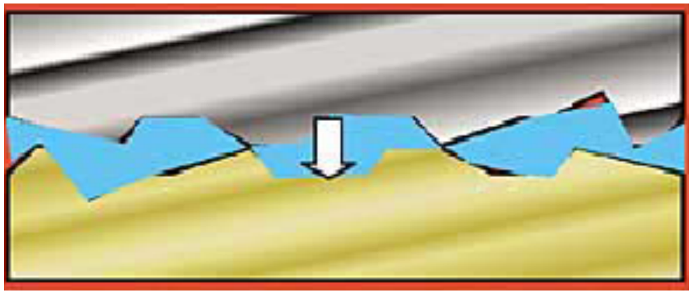
Nanotube fuzzy fibers as TIM



- TIM are high thermal conductivity materials to fill or eliminate air gaps
- Requirements of TIM: conform to mating surface, good wettability, provide high conductivity path.
- Examples of TIM: greases, reactive compounds, elastomers and pressure sensitive adhesive films

Thermal Impedance to Characterize Interfaces

Thermal Interface Materials



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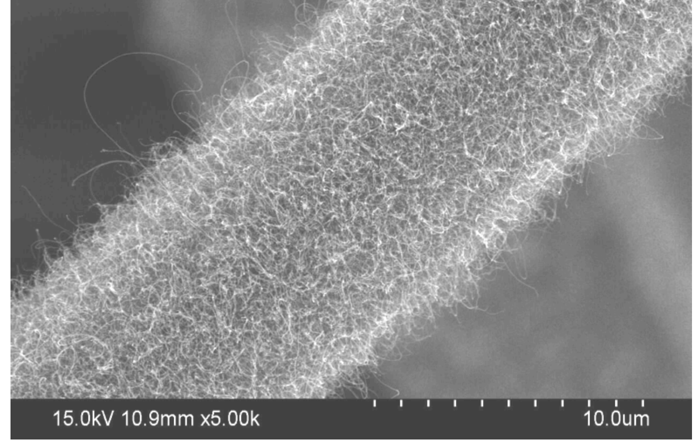
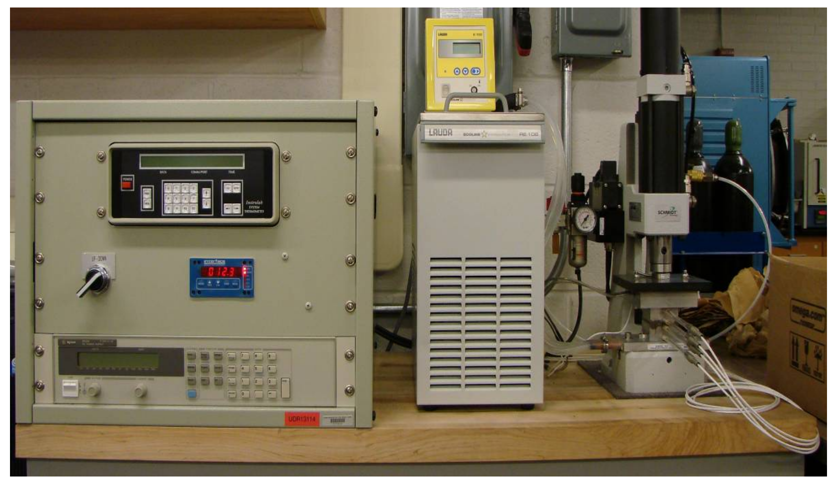


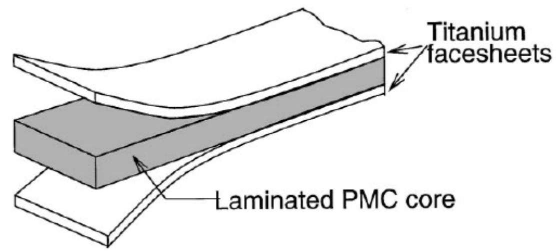
ASTM D5470 Thermal Interface Material Testing System

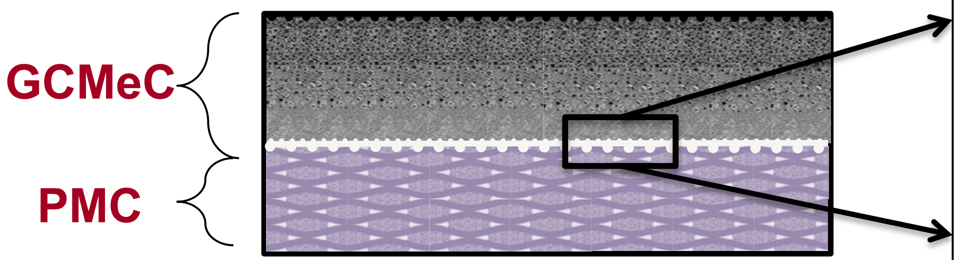


Delamination Resistant Interfaces

I. Benchmark by testing sample coupons of ***TiGr/Ti*** and ***TiGr/PMC***

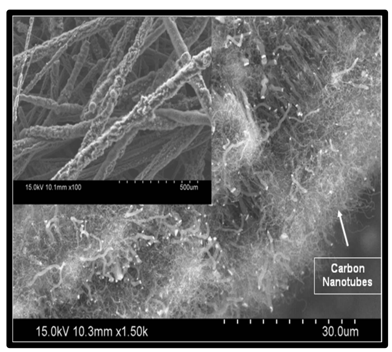
II. Evaluate ***GCMeC/PMC*** interface:





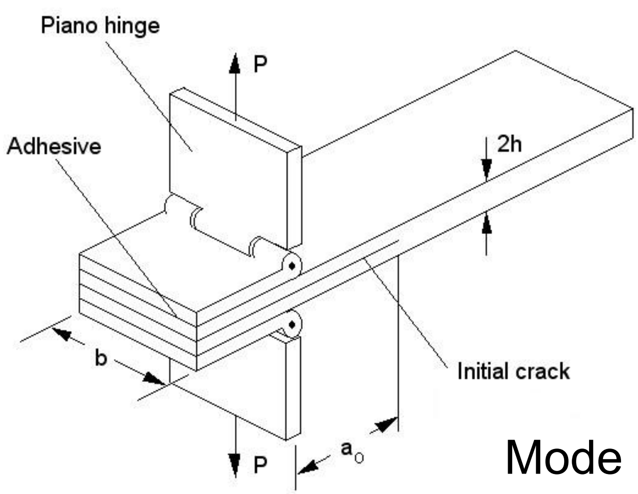
- *vertical nanocolumns followed by resin infusion*
- *bonding of metal to an intermediate fabric preform using vertical columns grown on both surfaces, with subsequent infusion of resin*
- *using Z-pinning technology*

Carbon nanotube grown on a Nickel wire using CVD. The inset shows a magnified image of the forest.

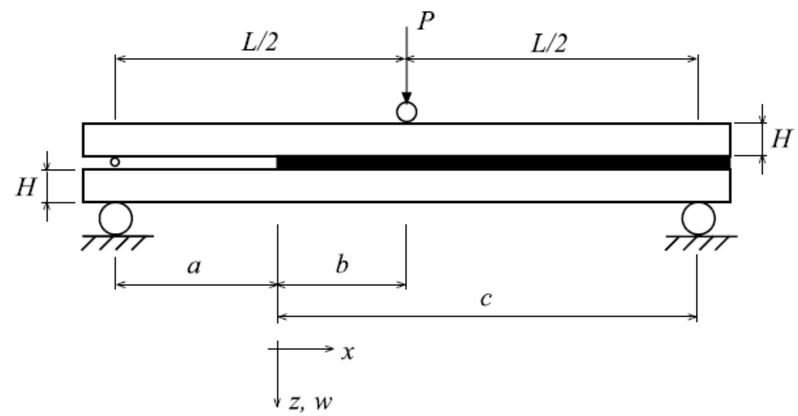


Quasi-static Interfacial Fracture Testing

- Assess interfacial integrity of GCMeC/PMC through the double cantilever beam (DCB) test for pure Mode-I loading and end notch flexure (ENF) test for pure Mode-II loading from room temperature to 1000 °C.



Mode I

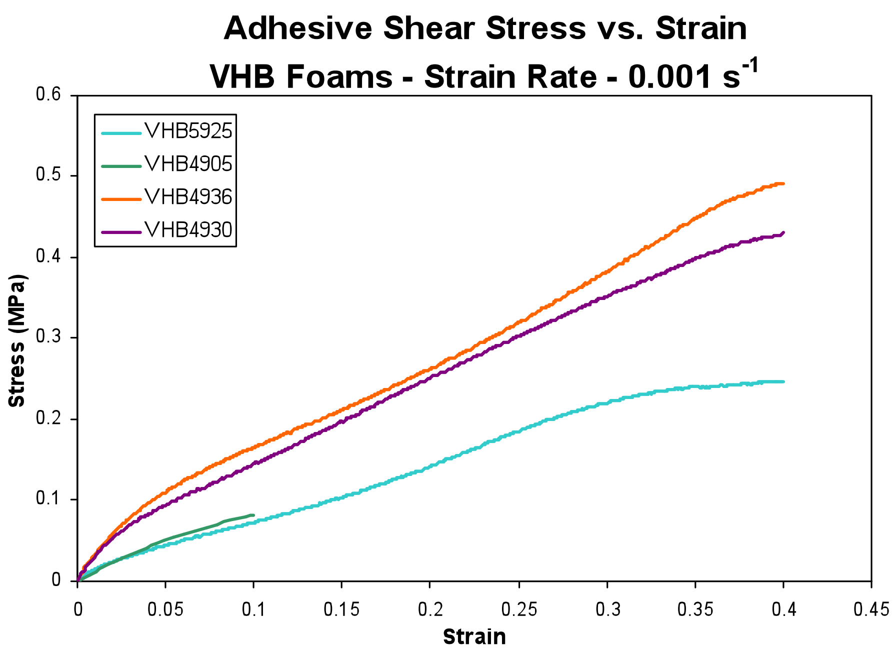


Mode II

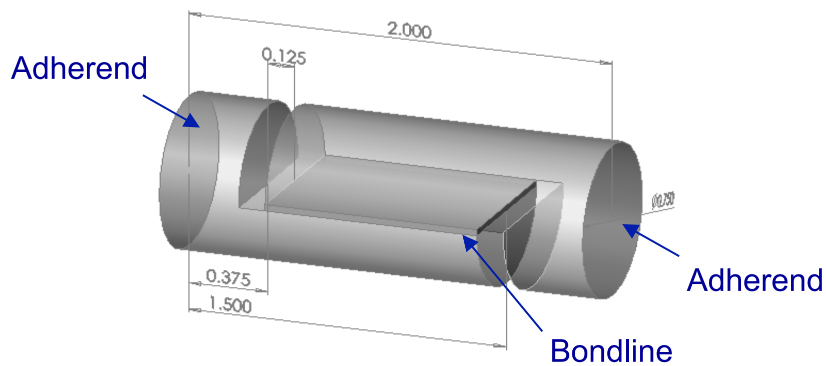
- Utilize SEM at different stages of the bending experiments to qualify the crack opening profile.
- Combined mesoscale characterization (SEM) and macro-scale characterization will provide insight in to the fracture process and qualify interfacial delamination for the three joint concept.

Dynamic Shear Strength of Bonded Interface

- Determine the dynamic shear strength of interlayer materials and joining mechanisms used in the assembly of hybrid composites.
- Determine the static and dynamic adhesive shear strength between functional layers will be determined as a function of component properties and process conditions.



Test specimen

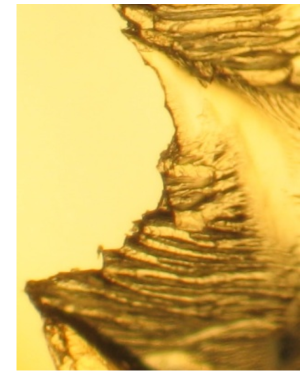


Structural Performance

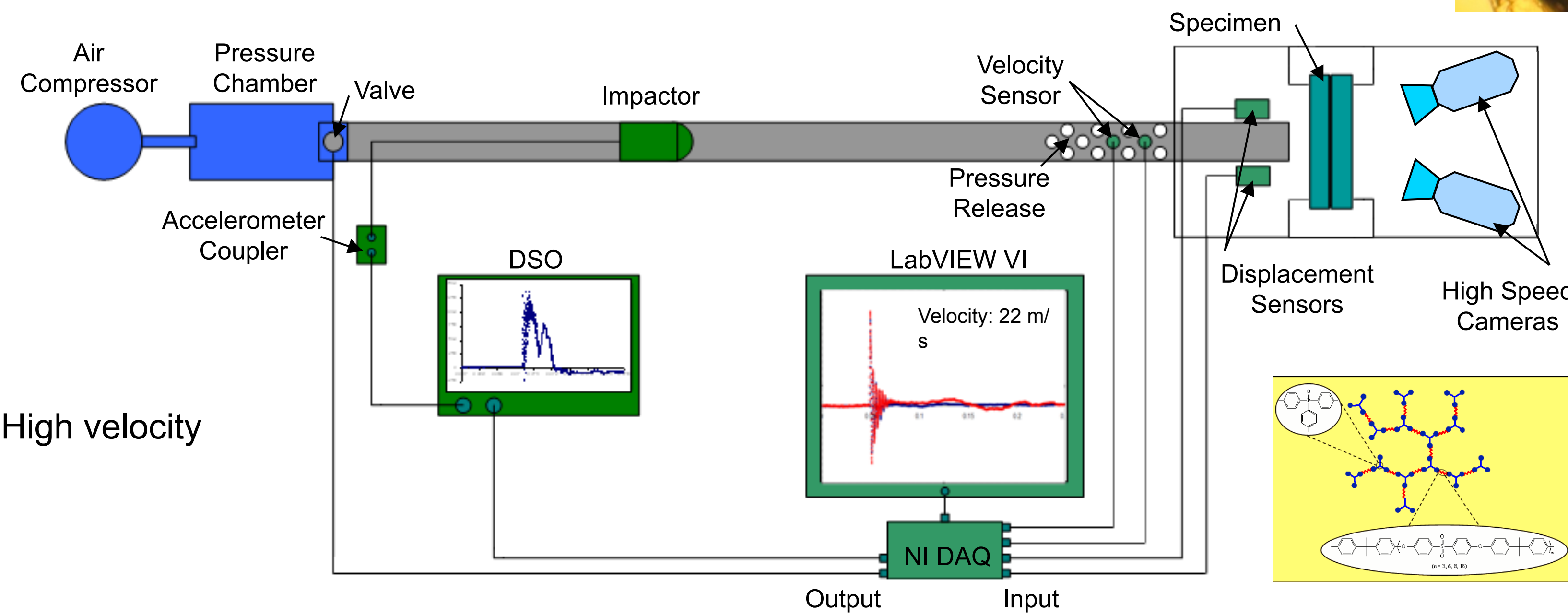
- **Impact Response**
  - Nakhiah Goulbourne (UM/VT)
- **Vibration Analysis**
  - Dan Inman (VT)

Impact Response Characterization

Effect of microstructure and functionalized assembly



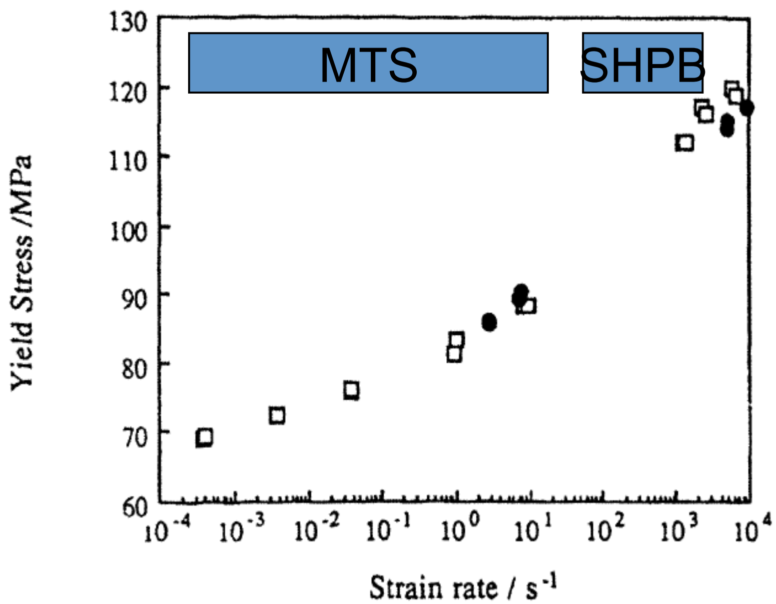
- Quasi-static, drop tower, and instrumented gas gun experiments
- Damage mechanisms and dominant failure modes will be identified and the potential for crack-arrest functionality of the component layers will be verified.
- Interactions at the interfaces will be of primary importance.

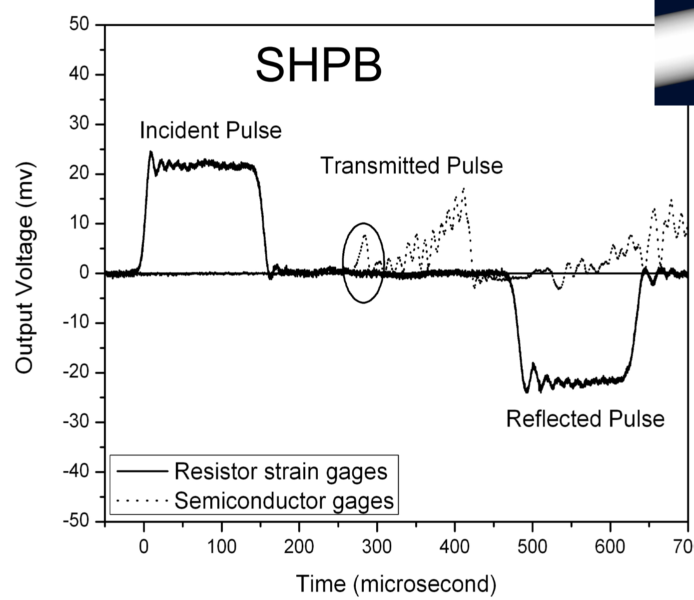


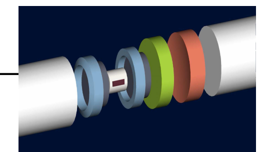
High velocity

Dependence of Mechanical Properties on Strain Rate

- Mechanical properties of the hybrid composites will be determined as a function of strain rate and a suitable constitutive formulation for the stress will be developed.
- For monolithic polymer components, correlations between microstructure and strain rate will be determined.
- For hybrid composites, correlations between joining technologies and strain rate dependence are sought.







Vibration Analysis

- State of the art vibration measurement systems will be used to measure time responses of FGHC macro samples
- Frequency response functions and time domain methods will then be used to determine the global modulus and damping parameters of the samples
- These experiments will be designed to verify and/or modify the results of the multiscale modeling effort across various temperatures and pressures of flight

VT altitude chamber capable of vibration measurements from -70° C to 170°C at pressures across the range of flight from sea level to 100,000 ft

Characterization Scales

nm

reinforcement/ matrix interface

µm

microvascular channels

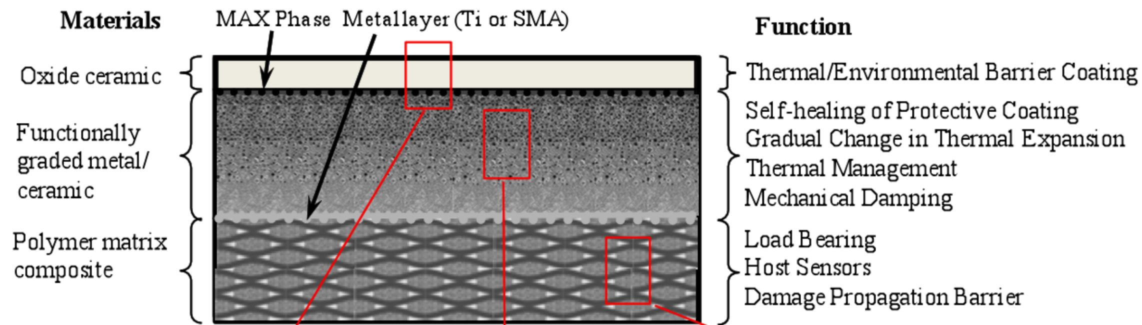
bonded interfaces

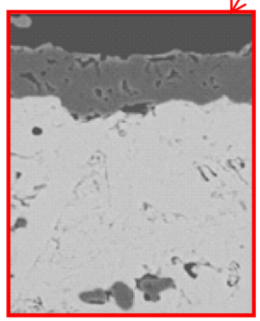
mm

composite layer properties

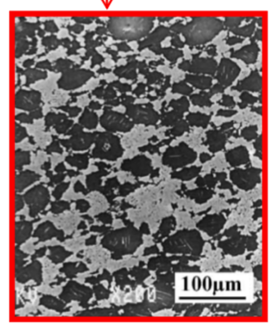
structural response

cm





SEM micrograph of 15 µm thick protective Al_2O_3 surface layer formed after 10,000 heating cycles of Ti_2AlC [Ref]



SEM of Ti_2AlC (light) + $\gamma TiAl$ (dark) as an example of MAX phase composite. [Ref]